MECHANICAL PROPERTIES OF A NEW ZINC-REINFORCED GLASS IONOMER RESTORATIVE MATERIAL.

By

Sarah Sultan Al-Angari

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Melvin Lund

Michael A. Cochran

Tien-Min Gabriel Chu

Jeffrey A. Platt

Anderson Hara

N. Blaine Cook
Chairman of the Research Committee

Date

DEDICATION

First I would thank Allah, who helped me to accomplish this thesis.

I dedicate this thesis to my father, mother, husband, my two kids, sisters and brothers and family a great source of motivation, inspiration, and support. A special thanks to my dearest friend Dr. Alaa Sabrah who was there for me the whole time.

May God bless them all.

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INTRODUCTION

Glass ionomer cements (GIC) were introduced in 1969 by Wilson and Kent.[1] They are "composed of a calcium fluoroaluminosilicate glass powder and an aqueous solution of an acrylic acid homo- or copolymer (polyelectrolyte)".[2] Commercially available glass ionomers can be divided into two major categories, conventional glass ionomer cements (CGIC) and resin-modified glass ionomer cements (RMGIC).[3]

They are the restorative material of choice in many clinical situations due to their unique properties including tooth structure adhesion, fluoride release, coefficient of thermal expansion similar to the tooth structure and biocompatibility.[2] Despite these beneficial characteristics, CGICs have some less desirable physical and mechanical properties such as a relatively rough surface texture, high opacity and susceptibility to dehydration and moisture contamination during the initial setting .[4] They also have low fracture toughness and flexural strength,[5] which make them unsuitable for use in high-stress bearing areas, such as Class I and II restorations.[2, 6-9] Therefore, these materials have been most commonly used in low stress bearing areas such as Class III and Class V restorations, which may include anterior teeth and aesthetic zones.[10, 11]

The surface characteristics of GICs are important. Restorative materials are constantly subject to thermal, mechanical, and chemical challenges in the oral cavity leading to softening and surface roughness.[9, 12] Surface texture includes many aspects such as: wear, roughness and cracks. An increase in roughness might result in faster colonization and maturation of dental plaque, thereby increasing the risk of caries.[13] Wear, discoloration and surface roughness may all impair restoration shape, contour and aesthetics, [9] which can impact the clinical deterioration of restorative materials.[14]

While some restorative materials, such as ceramics, change minimally during wear in the oral cavity, GICs suffer degradation due to mechanical and/or chemical interaction with the oral environment.[9, 12, 15] Toothbrushing is one of many procedures that has an effect on dental materials. Daily brushing with dentifrice can gradually cause a change of color, loss of contour and roughening of the restoration surface.[12] Many studies have investigated GIC wear and surface microhardness after toothbrushing and concluded that there was less wear of CGICs when compared to RMGICs and that toothbrushing can cause a decrease in the microhardness and an increase in both abrasion and surface roughness, but varies with the material.[16-19] Also when compared to composite resins, GICs had higher surface roughness and wear as well as lower hardness.[16, 17, 20]

Microhardness is regarded as an important property to predict the clinical performance of restorative materials.[21] Hardness is the resistance to permanent indentation on the surface of a solid material when a force is applied. It relates to functional parameters of a restorative material such as wear and the ability to withstand fracture.[22] Studies have shown that microhardness in GIC restorations is higher in CGICs compared to RMGICs [2, 23] and when a combination of smaller glass particles and less porosity were incorporated in the glass ionomer material. [12, 21, 24]

Fracture toughness, is an intrinsic property of a material which measures the energy required to propagate a crack from an existing defect.[25, 26] It is a useful indicator of resistance to marginal fracture and wear.[27] Some studies done on fracture toughness of GICs show that there is no significant difference in the K_{Ic} values for glass ionomer-

based materials,[25, 26, 28] whereas, other studies show a significant amount of difference.[29, 30]

Recently, a new zinc-reinforced glass ionomer (ZRGI) restorative material was introduced (ChemFil Rock, Dentsply Caulk) and is claimed to have improved properties including hardness, wear resistance and fracture toughness. It is thought to be up to 25% stronger than other glass ionomer materials. However, further investigation is needed since limited data exist for this material in the literature.

The objectives of this in-vitro study were to compare four properties of the ZRGI: fracture toughness, abrasive wear, roughness and surface microhardness, to three commercially available GICs: a resin-coated glass ionomer (EQUIA Fil) and two high strength (packable) GICs (Fuji IX GP Extra and Ketac Molar Quick Aplicap). Premise resin-matrix composite (Kerr) was included as a control group (Table 1).

HYPOTHESES:

Null hypothesis: ZRGI would not demonstrate significantly higher fracture toughness, lower abrasive wear, lower roughness and higher surface microhardness than the three other GICs evaluated in this study.

Alternative hypothesis: ZRGI (ChemFil Rock) would demonstrate significantly higher fracture toughness, lower abrasive wear, lower roughness and higher surface microhardness than the three other GICs evaluated in this study.

REVIEW OF LITERATURE

BACKGROUND

Commercially available glass ionomer dental cements were first launched in Europe in 1975[31] and marketed in the United States in 1977. The original product was called ASPA (**A**lumino-**S**ilicate **P**oly-**A**crylate) and consisted of an ion-leachable aluminosilicate glass in an aqueous solution of a copolymer of acrylic acid.[1] But the material had inferior properties and characteristics compared to other restorative materials, such as higher roughness, lack of stability in the oral environment, difficulty in handling and poor esthetics.

Since then, many researchers have modified GIC composition by changing the particle size, shape and number, and incorporating amino acid derivatives and monomers such as NVP (N-vinylpyrrolidone) in to GIC.[30] The latest was to introduce nanofillers into RMGIC[12] to improve mechanical properties and aesthetics.

GENERAL COMPOSITION

GIC are composed of a calcium fluoroaluminosilicate glass powder (SiO_2-AlO_3-CaF_2-$AlPO_4$-$NaAlF_6$) and an aqueous solution of an acrylic acid homo- or copolymer (polyelectrolyte).[2] Commercially available GIC can be divided into two major categories, conventional glass ionomer cements (CGIC) and resin-modified glass ionomer cements (RMGIC).[3]

Chemical studies on the setting reaction of the conventional GIC describe an acid-base reaction that occurs when the powder and liquid are mixed together. The first phase of the setting reaction consists of the formation of a continuous calcium aluminosilicate matrix with partly crystalline calcium fluoride-rich droplets. The

concentration of the droplets is mainly dependent on how the glass particles were treated thermally.[32] The second phase of the setting process takes place when the glass is mixed with poly (acrylic) acid. “This process has two overlapping stages depending on the rapid leaching of calcium ions from the uncrystalline part of the droplets, followed by the slower release of aluminum (and some calcium) from the main glass phase.”[32] Setting is affected by two major factors, the microstructure and microcomposition of the glass.[32] Studies have shown that incorporating amino acid derivatives and monomers such as NVP (N-vinylpyrrolidone) into the GIC[30] or introducing nanofillers into RMGIC[12] which consists of small glass particles (nano-sised powder particles and fluorapatite) that may improve both physical and mechanical properties.

HISTORICAL DEVELOPMENT OF GIC:

The use of GIC restorations has expanded since they were introduced to the dental profession leading to the different GIC materials available today. After introducing ASPA in 1975, several changes have been incorporated in the material to enhance its mechanical and esthetic properties.

CGIC has been used by dentists because of its biocompatibility, low cytotoxicity, [33] fluoride release,[34] coefficient of thermal expansion being similar to tooth structure, low microleakage and ability to bond to the tooth structure. [2, 5, 18]

In spite of the improvements to GIC, its use has been limited to a few clinical situations due to its shortcomings including: “high sensitivity to changes in matrix water content during the setting reaction,”[35] low strength, inferior esthetic properties

when compared to composite,[36] high brittleness, and low fracture toughness and flexural strength.[2, 5]

In the late 70's, a GIC called Cermet was introduced where the glass particles were sintered with silver in an attempt to make GIC more useful in high stress bearing areas but no clinical improvement was shown.[37] In the mid to late 1980's, RMGIC was developed in an attempt to improve mechanical properties. They are a hybrid of two materials, GIC and composite resin, and thus contain acid-base and polymerizable reaction components. They have superior esthetic properties,[38] are higher in strength, are less technique sensitive and much more easily handled than CGIC.[25, 39] On the other hand, conventional GIC have comparable or greater fluoride release and rechargability, and better wear resistance when compared to RMGIC.[9, 16, 34]

MICROHARDNESS

"Microhardness is regarded as an important property to predict the clinical performance of restorative materials."[21] "Hardness is the resistance to permanent indentation on the surface."[40] Zanata et al. relate it to functional parameters of restorative materials such as wear and the ability to withstand fracture.[22] Indentation on the surface of the restoration may produce cracks that initiate a fracture. The microhardness of GIC has been evaluated in many studies. Bonifacio et al. and Xie et al. both concluded that CGIC have higher hardness when compared to RMGIC.[2, 23] In a study done by De Paula et al., higher hardness values were seen when a combination of smaller glass particles and less porosity were incorporated in the glass ionomer

material. [12] Momoi et al., in a study comparing the hardness of CGIC, RMGIC and composite resin observed higher hardness values for the composite followed by CGIC and RMGIC.[16] Zanata et al. tested the hardness of high viscosity GIC for ten years and concluded that the hardness initially tends to increase but that after 180 days there were no significant change.[41]

ABRASIVE WEAR AND ROUGHNESS

Abrasion is a word derived from the Latin verb abradere (to scrape off).[42] It describes the wearing away of a substance or structure through mechanical processes, such as grinding, rubbing or scraping.[43] Surface texture of dental materials includes many aspects such as roughness, wear and cracks. It has an influence on plaque accumulation, wear and discoloration of restorations which may eventually impair shape, contour and aesthetics.[9]

Wear resistance is one of the most challenging properties of direct dental restoratives. In recent years, many clinical and in vitro test methods have been developed to evaluate abrasive wear of restorative materials, including different two-body wear, three-body wear testing machines with different modifications as well as many different tooth-brushing model devices.

Efforts have been made to improve GIC wear-resistance through incorporation of silver or amalgam particles or the addition of mont-morillonite clay filler.[44, 45] But, none of these efforts has shown great improvement in wear-resistance.

A study by de Gee et al. evaluated the long-term wear changes in conventional GIC, metal-reinforced GIC and RMGIC and concluded that because there were high

amounts of material wear, they are not recommended for use in high-stress-bearing situations.[9] Momoi et al., in an in vitro toothbrush-dentifrice abrasion test, compared conventional GIC to RMGIC and concluded that there was more surface wear of RMGIC when compared to conventional GIC and that all the materials tested were rougher after toothbrushing.[16] Frazier et al. compared the toothbrush wear-resistance of RMGIC, compomers and resins and showed that there was no correlation between wear-resistance and the filler content.[18] Bala et al. examined the hardness and surface roughness of different kinds of GIC; GIC with nanofiller, silver-reinforced GIC, RMGIC and conventional GIC. There were significant differences among some materials that were attributed to the difference in composition; size of particles and type of fillers. Nanofilled GIC had the smoothest surface, whereas silver reinforced GIC had the highest microhardness value.[24] A study by Rios et al. compared the toothbrushing wear and roughness of GIC when used as sealants, and concluded that GIC with flowable consistency (Fuji Plus) had more wear and roughness than other restorative GIC (Ketac Molar ,Vitremer). In addition, restorative GIC had similar wear resistance but increased roughness when compared to resin-based sealants.[46]

FRACTURE TOUGHNESS

Fracture toughness is " The resistance of a material to brittle fracture when a crack is present in (or at the surface of) the material." It measures the amount of energy absorbed during propagation of a crack."[40] The clinical strength of a dental restorative material is better indicated by fracture toughness than average stress-based tests such as flexural strength.[47]

A study by Bonilla et al. tested fracture toughness of different core build up materials and concluded that all GIC are considered most prone to fracture.[25] Moshaverinia et al. compared the fracture toughness of a CGIC and a GIC containing N-vinylcaprolactam (NVC) and observed that modification in the properties of the GIC by adding (NVC) resulted in an enhancement of the fracture toughness values when compared to a conventional GIC.[30] Yamazaki et al. compared the fracture toughness of both CGIC and RMGIC and concluded that although RMGIC showed higher fracture toughness values than the CGIC, there was no statistical difference among the GIC.[28] Kovarik et al. examined fracture toughness of a composite and different GIC and concluded that there are significant differences in the fracture toughness values of GIC and that RMGIC had the highest fracture toughness values.[29]

MATERIALS AND METHODS

STUDY DESIGN

This study was conducted in two parts. The first compared microhardness, toothbrush abrasion, and surface roughness of four GIC and a composite resin as a control (Table 1). Part two tested fracture toughness of the four GIC listed in Table 1. For consistency, shade A2 of all materials was used.

MICROHARDNESS, ABRASIVE WEAR AND SURFACE ROUGHNESS

Specimens of ChemFil Rock, Fuji IX GP Extra, Ketac Molar Quick Aplicap, EQUIA Fil and Premise (n=9) were fabricated for a total of 45 specimens. The glass ionomer materials were mixed in a ProMix amalgamator according to manufacturer instructions (Table 1) and injected into circular metal molds (Ø= 5mm, 2mm in height). (Figure 1) The surface was covered with a Mylar strip and a glass slide with pressure applied manually to express excess material to create a flat surface. The specimens were allowed to set according to the manufacturer instructions. (Table 1) For EQUIA Fil, its accompanying resin surface sealant was applied and light cured for 20 seconds according to manufacturer instructions. The composite was injected into the metal molds and covered with a Mylar strip and glass slide in the same manner as glass ionomer, then polymerized using the Demi light-curing unit (Kerr, Danbury, CT, USA) with an 11 mm diameter light tip through the glass slide for 40 seconds. The specimens were maintained in 100% relative humidity at 37 degrees C for 20 minutes.[16, 48] Then each was embedded in acrylic resin (Varidur, High Performance Mounting Kit,

Buehler) in a block mold with dimensions of (12.25mm width × 12.5 length × 8.25 mm height) to facilitate mounting in the testing devices. (Figure 2) The testing surface of each specimen was ground flat to the level of the mold [49] and wet- polished using a sequence of 500,1200, 2400 and 4000 grit silicon carbide paper,[17, 50] then immersed in distilled water at 37 degrees C for 24 hours.[24, 48-53]

The 45 specimens were randomly divided into 3 testing groups of 15 specimens with 3 specimens of each material in each group. All tests were completed on the specimens of one group before proceeding to specimens of the next group.

Each specimen surface was scanned by a non-contact 3D optical profilometer (Proscan 2000, Scantron, Taunton England), (Figure 3) using the S5/03 chromatic sensor. Two areas were scanned, one 0.5 × 0.5 mm on the central area for roughness measurement with a step size of (0.1 × 0.1) and another 3 × 3mm involving the whole specimen surface for the determination of surface wear with a step size of (10×10). The scans were done at 100 Hz frequency, with full sensor speed (100%) and were considered as the baseline for calculations of surface loss and surface roughness changes.

Microhardness test:

Prior to wear testing, microhardness measurements were made around the periphery of the surface of each specimen using a Knoop hardness tester, Leco LM247AT (LECO Corporation, MI, USA), (Figures 4 and 5).

After positioning the specimen (Figure 6) on the testing machine, a diamond indenter with 25 gram load and 30 second dwell time[2, 23, 54] tested three points on the surface for

each specimen. The average of the three measurements (indentations) was considered as the microhardness value for the specimen (Figure 7, 8, 9, 10, 11).

Abrasive wear and surface roughness testing:

The specimens were then positioned on a custom-made toothbrushing machine. The specimen surfaces were brushed using a straight, soft toothbrush (Oral-B 40) fixed in the toothbrush holder of the machine (Figure 12) so that all the bristles were in contact with the specimen surface. Testing was done under 200 grams of load,[12, 18, 46] at a speed of 175 cycle/min for 20,000 double strokes[13, 16, 55] simulating two years of brushing.[13] A dentifrice (Crest Cavity Protection) was used as an abrasive slurry with a paste to water ratio of 1:1.[13, 50, 55] Each vessel of the machine was loaded with 80 grams of the slurry (Figure 13). The toothbrush and slurry were replaced after the testing of each specimen.

Following the toothbrush abrasion test, the specimens were rinsed with tap water and gently air dried. Both surface roughness and the amount of vertical material loss were measured using the optical profilometer with the scanning settings previously described. Specimens were positioned on the machine so that the measurements were recorded as the detector moved across the specimen surface area perpendicular to the direction of the toothbrushing movements (Figure 14). The surface loss results were calculated by image subtraction considering the baseline scans, using dedicated software (version 2,0,17 Scantron Industrial Products Ltd., Taunton, England) (Figure 15).

FRACTURE TOUGHNESS (K_{Ic})

Fracture toughness was determined according to ISO 13586, using 10 single-edge notched-bend specimens of each glass ionomer material (total of 40 specimens). The specimen dimensions were (25 mm length, 2.5 mm height, 5 mm width), with a notch of 2mm (Figure 16). Specimens were fabricated by mixing the respective glass ionomer materials according to manufacturer instructions and injecting into the metal mold. The top of each specimen was covered with a Mylar strip and a glass slide with pressure applied manually to remove excess material to create a flat surface. Each specimen was allowed to set according to the manufacturer instructions, removed from the mold then all sided of the specimen were finished using 320 and 400 grit silicon carbide paper. EQUIA Fil was coated with a surface sealant covering all surfaces of the specimen and polymerized for 20 seconds on each side using the Demi light-curing unit (Kerr, Danbury, CT, USA). Specimens were maintained in 100% relative humidity for one hour,[28] then in distilled water at 37°C for 24 hours prior to testing.[30, 48]

Before fracture toughness testing was conducted, the specimen height and width were measured using a digital caliper (Mitutoyo, Corporation, Japan). Those values were entered for each specimen into the TestWorks 4 software (MTS) associated with the screw-driven universal testing machine (MTS Sintech Renew 1123,Eden Prairie, MN) (Figure 17). Using a three point bending test device and a crosshead speed of 0.2 mm/min at room temperature, the specimens were loaded to fracture (Figure 18-19).

Fracture of the specimen was identified by a sudden drop in load during the test. Fracture toughness (K_{Ic}) was calculated from the following equation:

$K_{Ic} = f(a/w)(F/h\sqrt{w})$

Where:

K_{Ic} Fracture toughness ($MPa \cdot m^{1/2}$)

F Force at the beginning of crack propagation (N)

a Crack length (mm)

h Specimen thickness

w Specimen width (mm)

$f(a/w)$ Fracture geometry factor ; which can be calculated by:

$$6\alpha^{1/2}[1.99 - \alpha(1-\alpha)(2.15 - 3.93\alpha + 2.7\alpha^{2})] / [(1+2\alpha)(1-\alpha)^{3/2}]$$

A material with a higher K_{Ic} value is more resistant to crack propagation than a material with a lower value.

STATISTICAL METHODS

Knoop hardness, abrasive wear, surface roughness (R_a) and fracture toughness (K_{Ic}) measurements were summarized (mean, standard deviation, standard error, range) for all of the materials.

The data were not normally distributed; therefore Wilcoxon Rank Sum tests were used with the results presented using box plots to compare the groups for differences in each outcome regarding Knoop hardness, abrasive wear , surface roughness and fracture toughness (K_{Ic}). A 5% significance level was used.

SAMPLE SIZE:

Based on previous studies,[23, 25, 56] the within-group standard deviation estimates for K_{Ic}, abrasive wear, and Knoop hardness were 0.07 MN · m-3/2, 6µm, and 11 MPa, respectively. With a sample size of nine per group, the study had a 80% power to detect differences between any two groups with a difference of 11 µm for abrasive wear and 20 KHN for Knoop hardness, assuming two-sided tests and an overall 5% significance level. With a sample size of ten per group, the study had a 80% power to detect differences between any two groups of 0.12 for K_{Ic}. The within-group standard deviation estimate for Ra was 0.25µm based on prior studies.[46, 57, 58] With nine samples per group, the study was able to detect 0.46 µm differences between groups for Ra.

RESULTS

KNOOP MICROHARDNESS RESULTS

The Knoop hardness measurements including the mean values and the standard deviations are summarized in (Table 2, Figure 20). Statistically significant differences in Knoop hardness were found among material groups ($p < 0.005$). The mean hardness values of Fuji IX GP Extra and Ketac Molar Quick Aplicap was significantly higher than that of EQUIA Fil. The mean value of ChemFil Rock was significantly lower than that of EQUIA Fil, Ketac Molar Quick Aplicap and Fuji IX GP Extra. Premise showed significantly lower mean hardness values when compared to the other groups.

Fuji IX showed the highest values (66.86 KHN), whereas Premise had the lowest values (45.44 KHN) among the tested materials.

TOOTHBRUSH ABRASION RESULTS

Table 3 and Figure 21 show the mean values and the standard deviations of surface loss resulting from the toothbrush abrasion test. The mean average surface loss values of ChemFil Rock, Fuji IX GP Extra, Ketac Molar Quick Aplicap, EQUIA Fil, and Premise were 4.69 µm, 5.21 µm, 3.79 µm, 5.72 µm and 3.07µm, respectively. No significant differences were found between the glass ionomer groups. EQUIA Fil, Fuji IX GP Extra and ChemFil Rock were significantly different from Premise. Ketac Molar showed intermediate surface loss, not differing significantly from any of the other groups.

ROUGHNESS RESULTS

The comparison of superficial roughness measurements (Ra) of each material before toothbrushing (baseline), after toothbrushing, the average roughness change and the standard deviations are presented in (Table 4, Figure 22) for all five restorative materials. The mean average change in roughness values of ChemFil Rock, Fuji IX GP Extra, Ketac Molar Quick Aplicap, EQUIA Fil, and Premise were 0.79 µm, 0.10 µm, 0.62 µm, 0.14 µm, 0.68 µm respectively. Significantly higher roughness change values were observed for ChemFil Rock when compared to Ketac Molar Quick Aplicap, EQUIA Fil and Fuji IX GP Extra. Premise showed intermediate change values, not differing significantly from any of the tested materials.

FRACTURE TOUGHNESS RESULTS

For all four materials tested in this portion of the study: ChemFil Rock, Fuji IX GP Extra, Ketac Molar Quick Aplicap and EQUIA Fil, examination of the fractured specimens revealed cracks that originated at the apex of the notch and progressed toward the load point. The fracture toughness mean and standard deviation measurements are shown in (Table 5, Figure 23).

EQUIA Fil, had the highest K_{Ic} value among the tested GICs with a mean of (1.21) MPa· $m^{½}$. This was significantly higher than ChemFil Rock, Fuji IX GP Extra and Ketac Molar Quick Aplicap. ChemFil Rock had mean values of (0.99 MPa·$m^{½}$) and was significantly higher than both Ketac Molar Quick Aplicap (0.85 MPa· $m^{½}$) and Fuji IX GP Extra (0.80 MPa·$m^{½}$), which did not differ from each other.

TABLES AND FIGURES

Table 1: Description of the tested restorative materials.

Material	Description	Manufacturer	Shade	Mixing Time (sec) / Setting Time (min)	Batch
ChemFil Rock	Zinc reinforced glass ionomer	Dentsply	A2	15 sec / 6:00	1105000887/ 1106000636
Fuji IX GP Extra	Packable glass ionomers	GC America	A2	10 sec / 2:30	1112101
Ketac Molar Quick Aplicap	Packable glass ionomers	ESPE	A2	10 sec / 3:30	471469
EQUIA Fil	Resin-coated glass ionomer cement	GC America	A2	10 sec / 2:30	1204241
Premise	Nanofilled Hybrid composite resin	Kerr	A2	N/A	4442265

Table 2: Mean and standard deviation summary of Knoop hardness (KHN) test.

Material	**n**	**Average KHN**	**SD**	*****
ChemFil Rock	9	52.39	2.67	a
Fuji IX GP Extra	9	66.86	5.36	b
Ketac Molar Quick Aplicap	9	62.53	2.91	b
EQUIA Fil	9	58.64	2.01	c
Premise	9	45.44	2.87	d

*Values with similar letters are not statistically different.

Table 3: Mean and the standard deviations of surface loss resulting from the toothbrush abrasion test.

Material	**n**	**Mean surface loss (µm)**	**SD**	*****
ChemFil Rock	9	4.69	1.23	a
EQUIA Fil	9	5.72	1.04	a
Fuji IX GP Extra	9	5.21	1.48	a
Ketac molar Quick Aplicap	9	3.79	2.82	ab
Premise	9	3.07	0.93	b

*Values with similar letters are not statistically different.

Table 4: Mean and standard deviation of surface roughness (Ra).

Material	**n**	**Mean Ra before (µm)**	**SD**	**Mean Ra after (µm)**	**SD**	**Mean Ra change (µm)**	**SD**	*****
ChemFil Rock	9	0.63	0.32	1.43	0.41	0.79	0.14	a
EQUIA Fil	9	1.13	0.76	1.27	0.80	0.14	0.46	b
Fuji IX GP Extra	9	1.16	0.85	1.26	0.45	0.10	0.98	b
Ketac molar Quick Aplicap	9	1.09	1.10	1.71	1.38	0.62	0.60	b
Premise	9	0.23	0.05	0.91	1.00	0.68	0.97	ab

*Values with similar letters are not statistically different.

Table 5: The fracture toughness mean and standard deviation measurements.

Material	**n**	**K_{IC} (MPa· $m^{½}$)**	**SD**	*****
ChemFil Rock	10	0.99	0.07	b
EQUIA Fil	10	1.21	0.23	a
Fuji IX GP Extra	10	0.80	0.04	c
Ketac molar Quick Aplicap	10	0.85	0.09	c

*Values with similar letters are not statistically different.

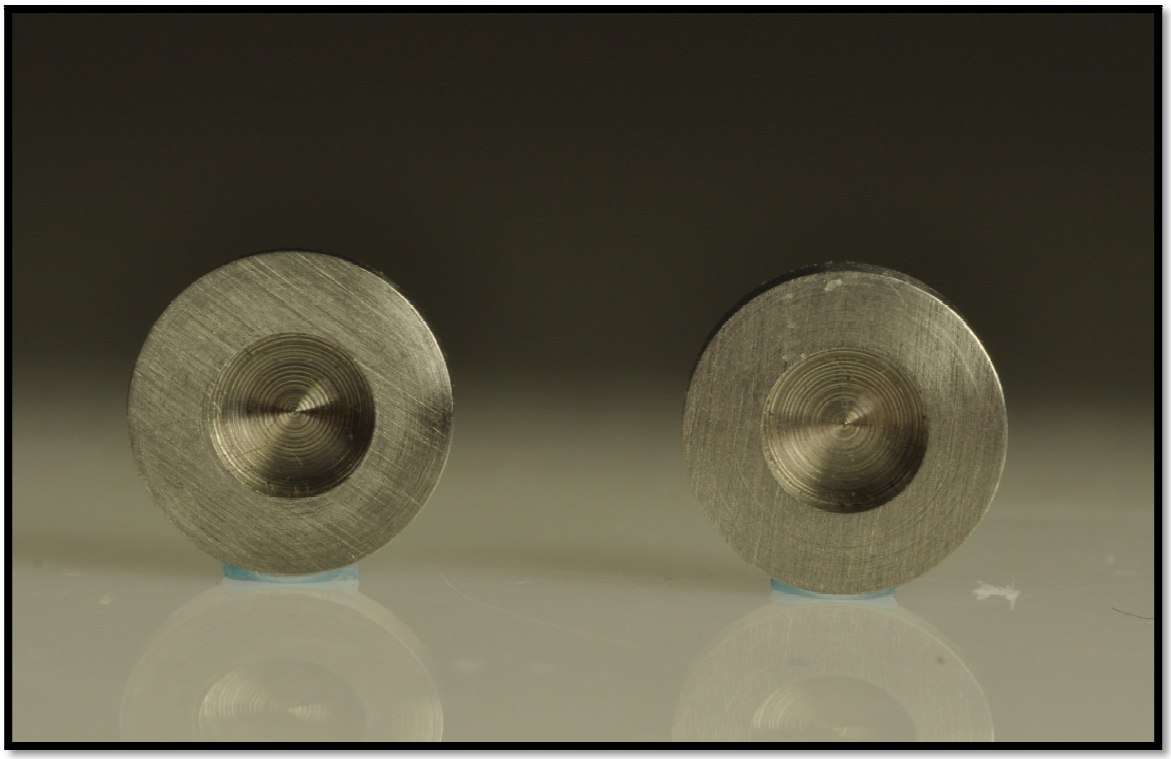


Figure1.Metal molds for abrasion, roughness and knoop hardness testing.

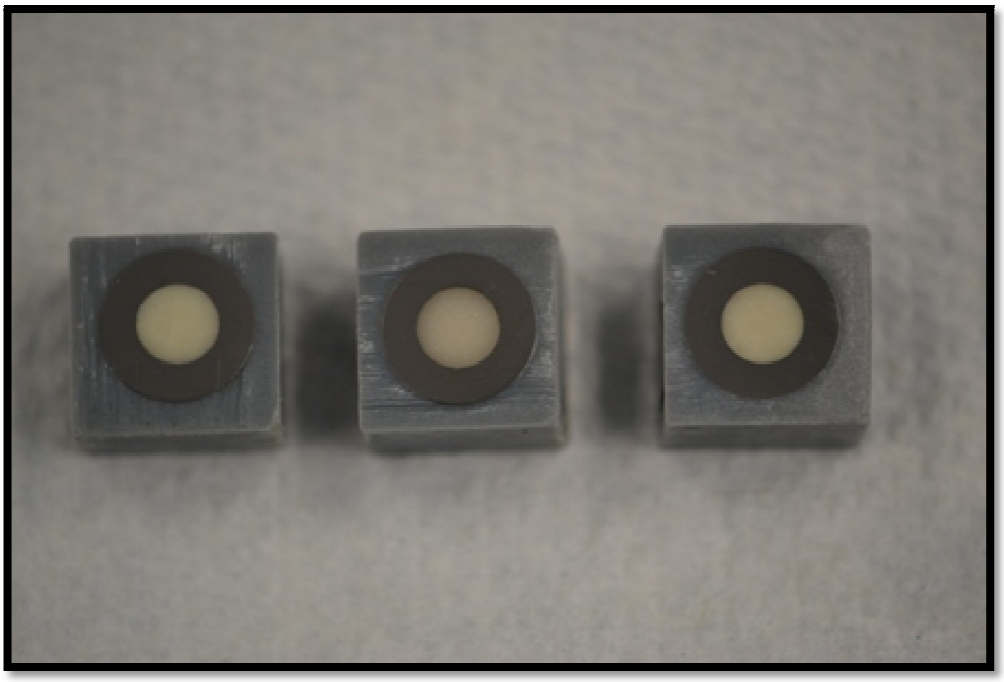


Figure 2. Specimen after preparation.

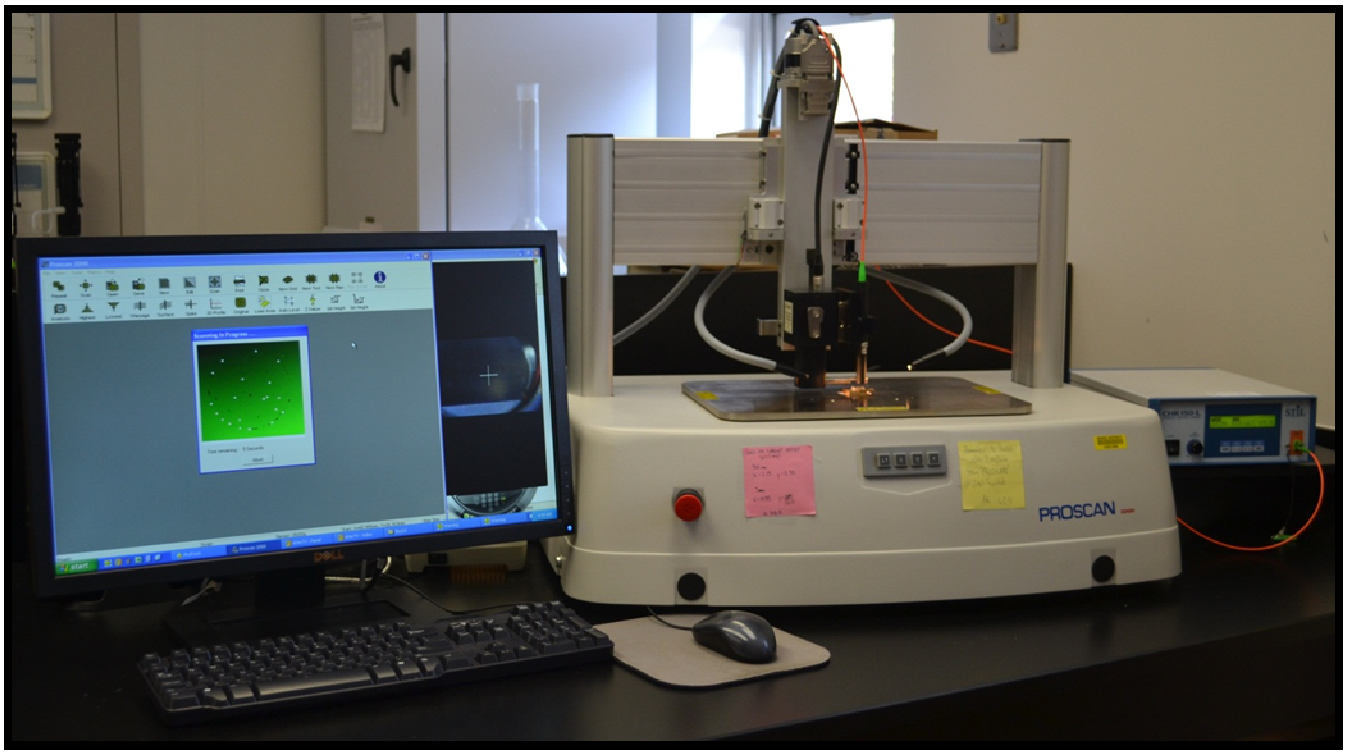


Figure 3. The optical profilometer (Proscan) machine.

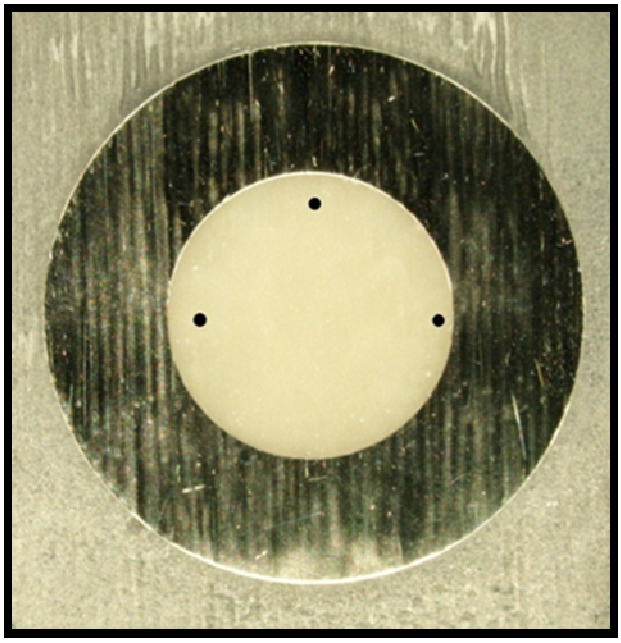


Figure 4.Specimen for abrasive wear, roughness and microhardness tests with the indentation locations.

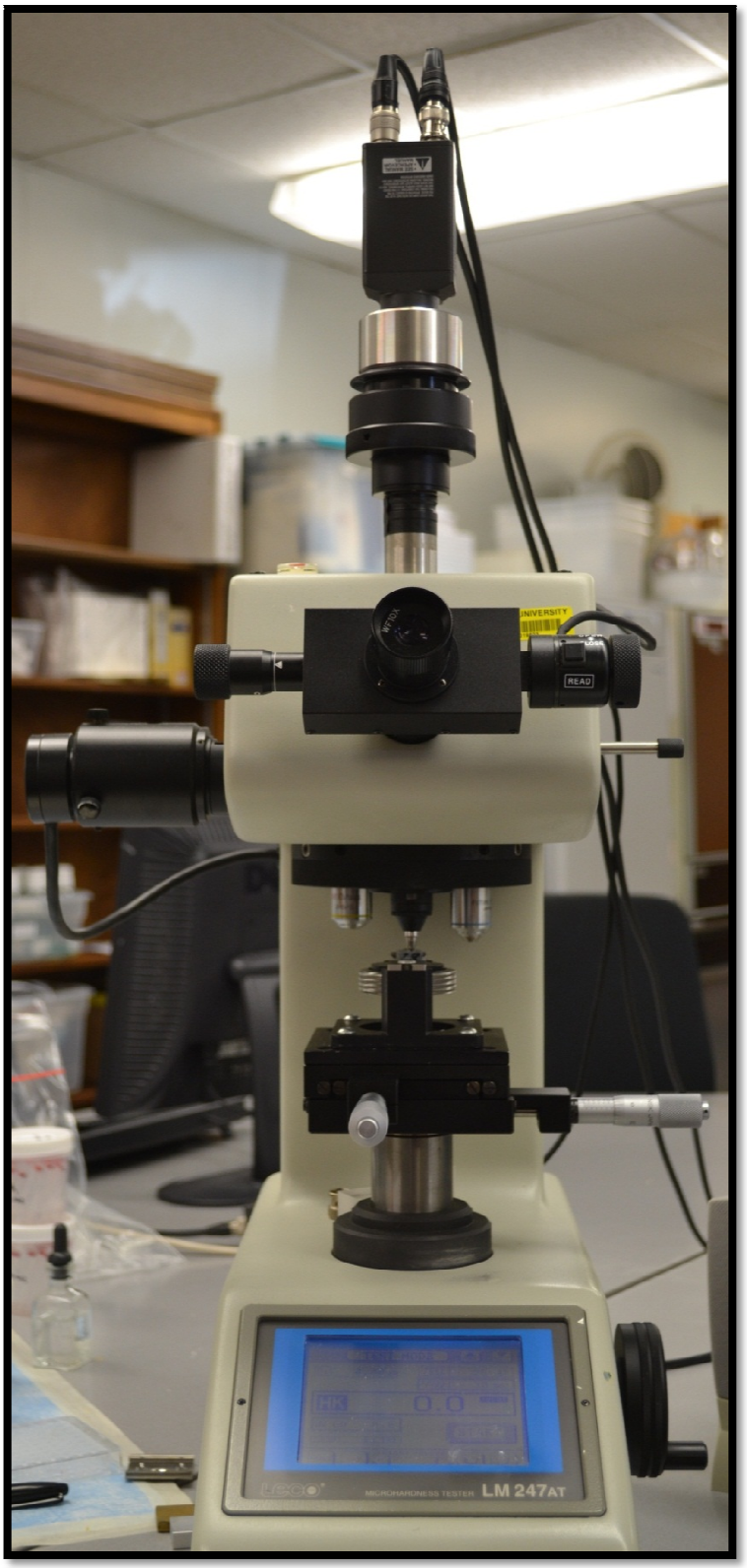


Figure 5.Knoop hardness machine.

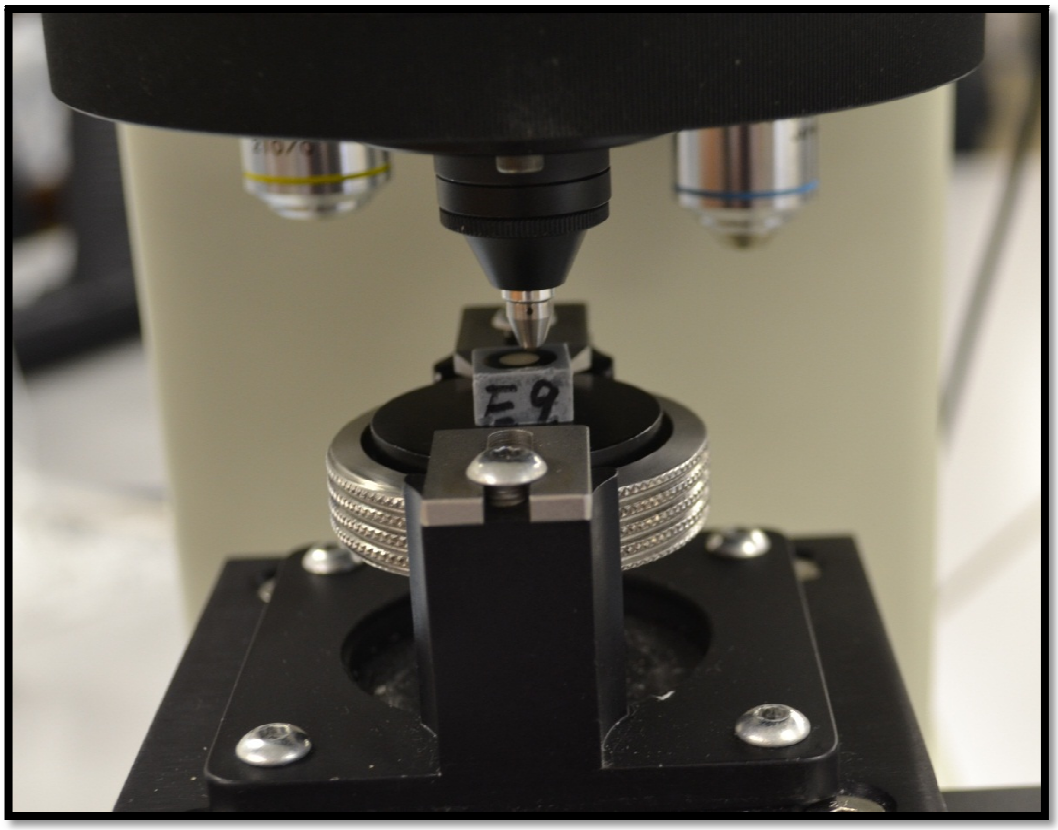


Figure 6. Specimen positioned on the Knoop hardness machine.

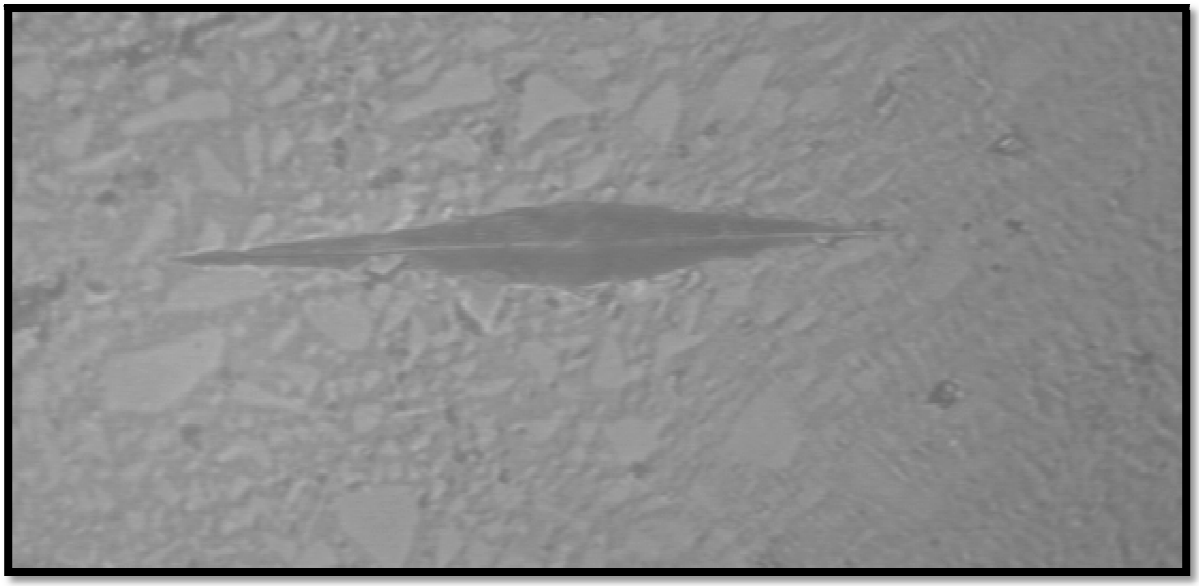


Figure 7. Microhardness indent on ChemFil Rock.

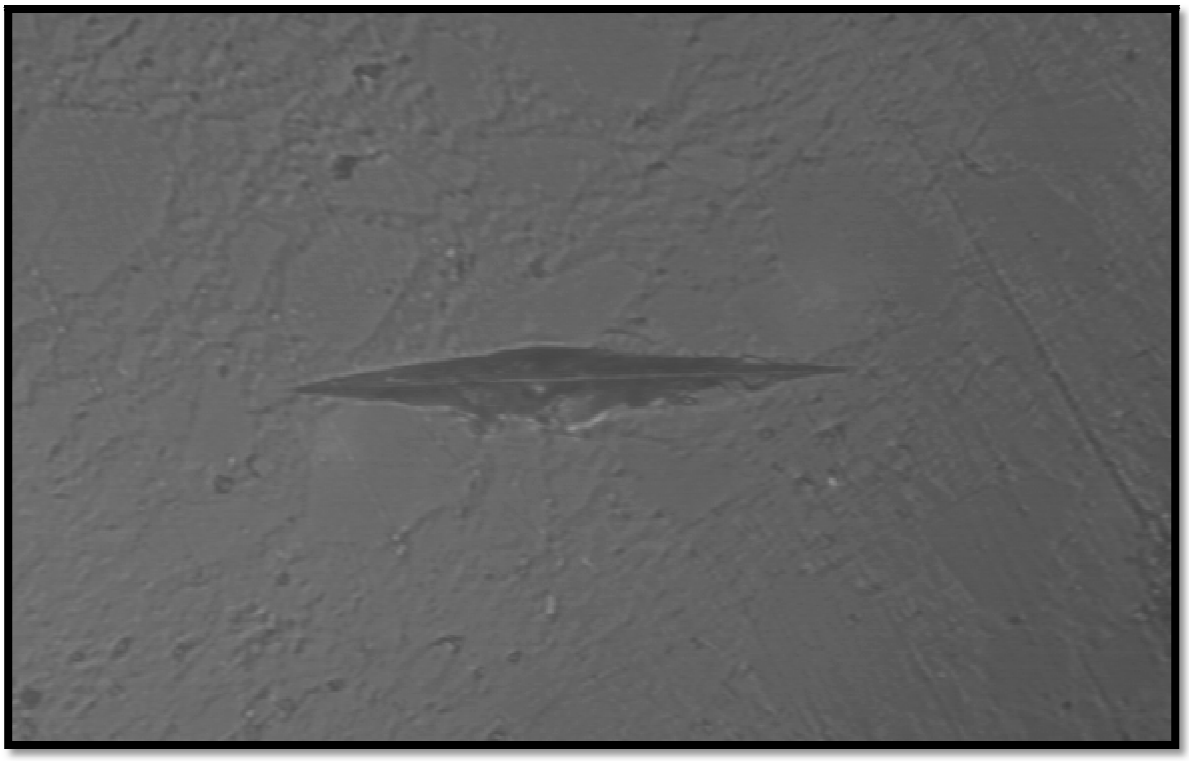


Figure 8. Microhardness indent on Fuji IX GP EXTRA

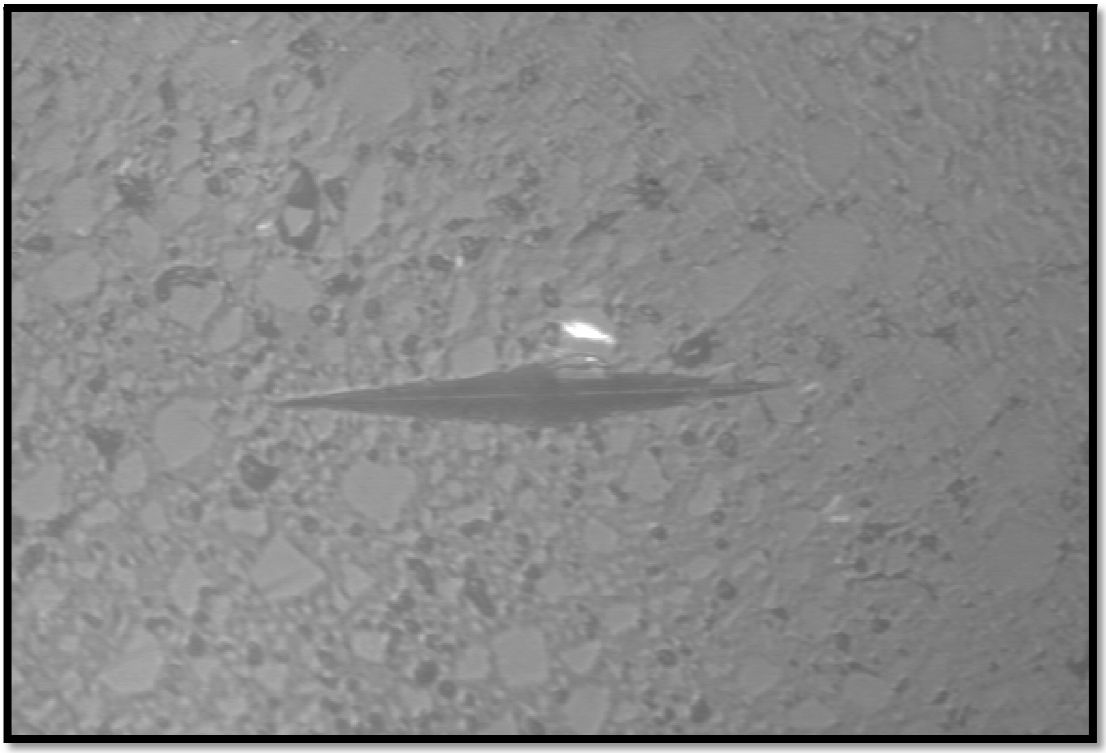


Figure 9. Microhardness indent on Ketac Molar.

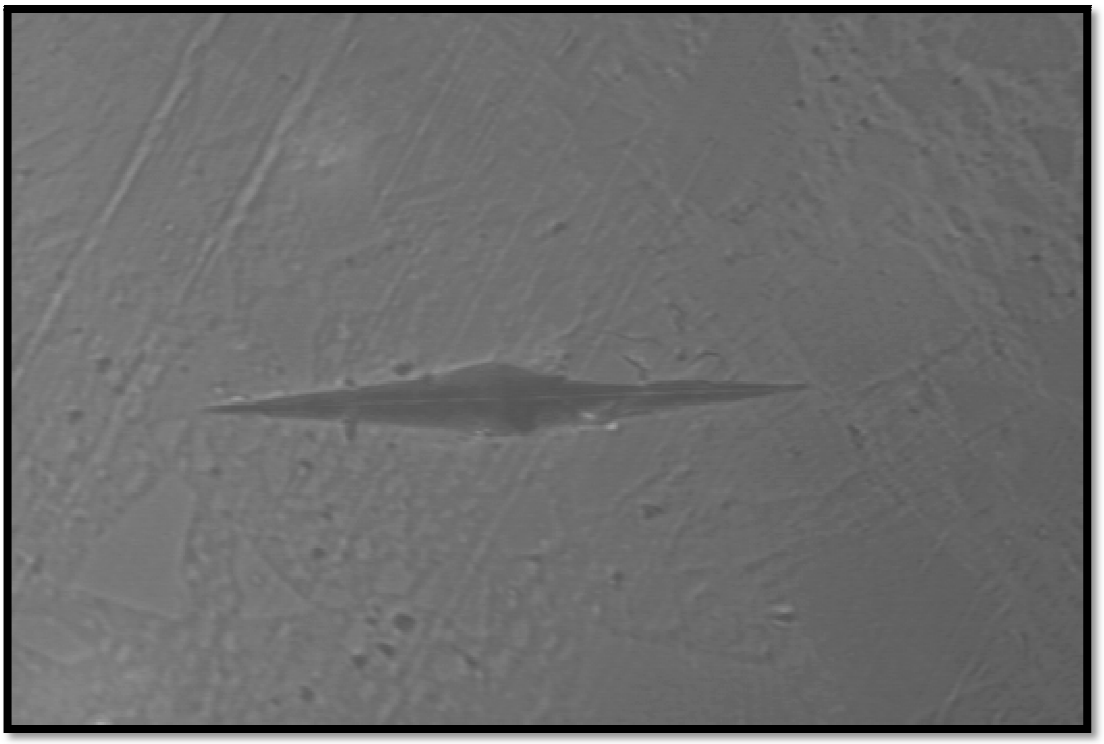


Figure 10. Microhardness indent on Equia Fil.

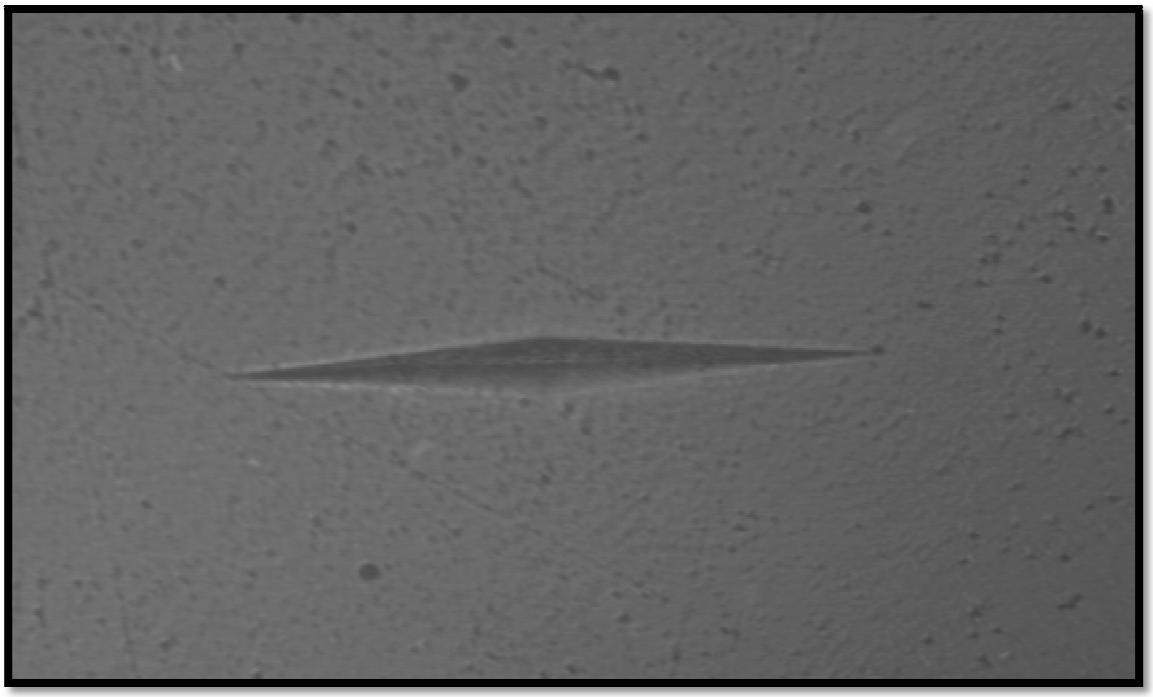
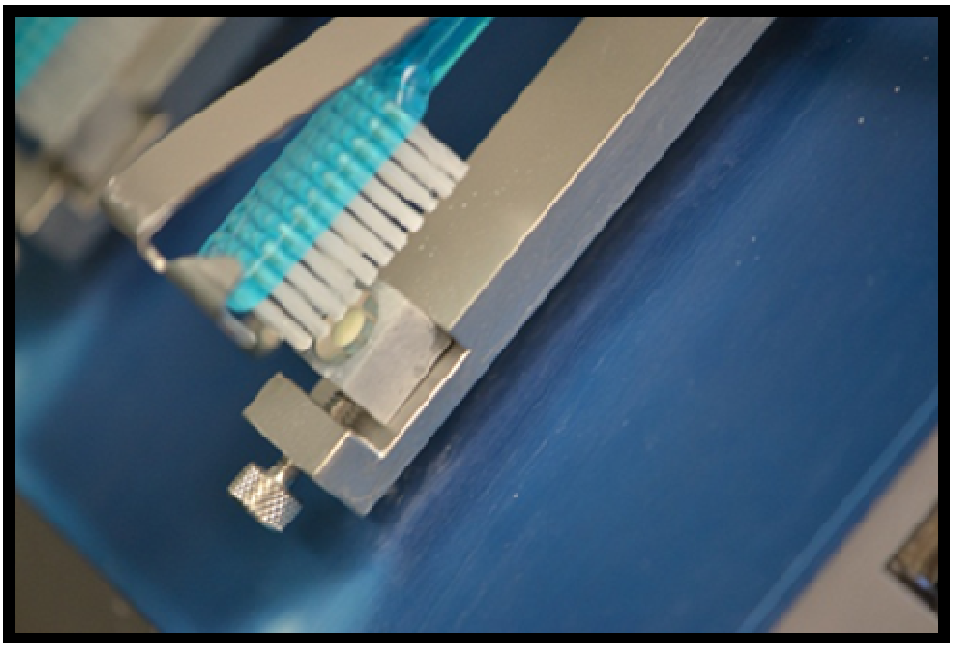


Figure 11. Microhardness indent on Premise.



(a)

(b)

Figure 12. (a ,b) Specimen positioned at toothbrushing machine.

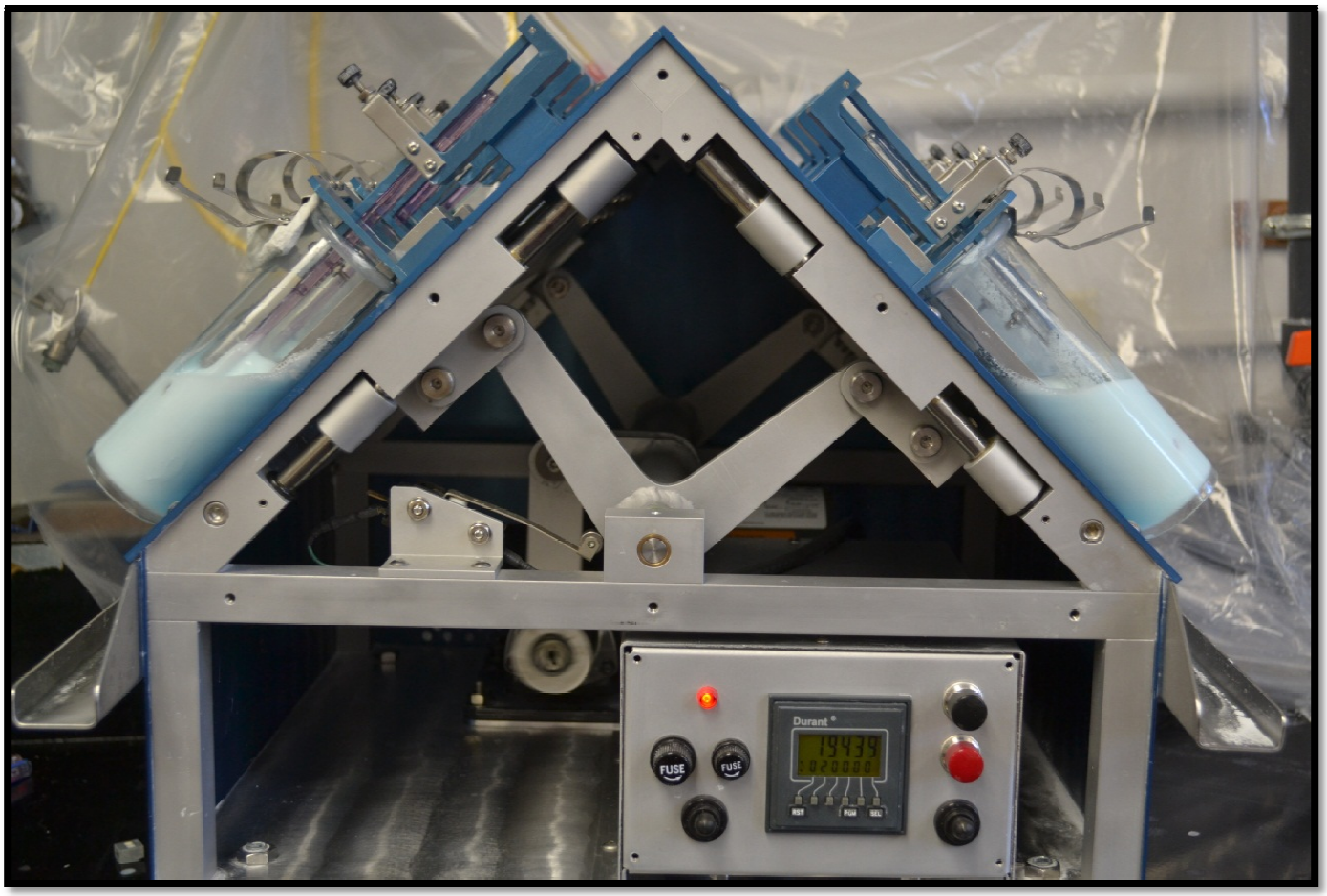
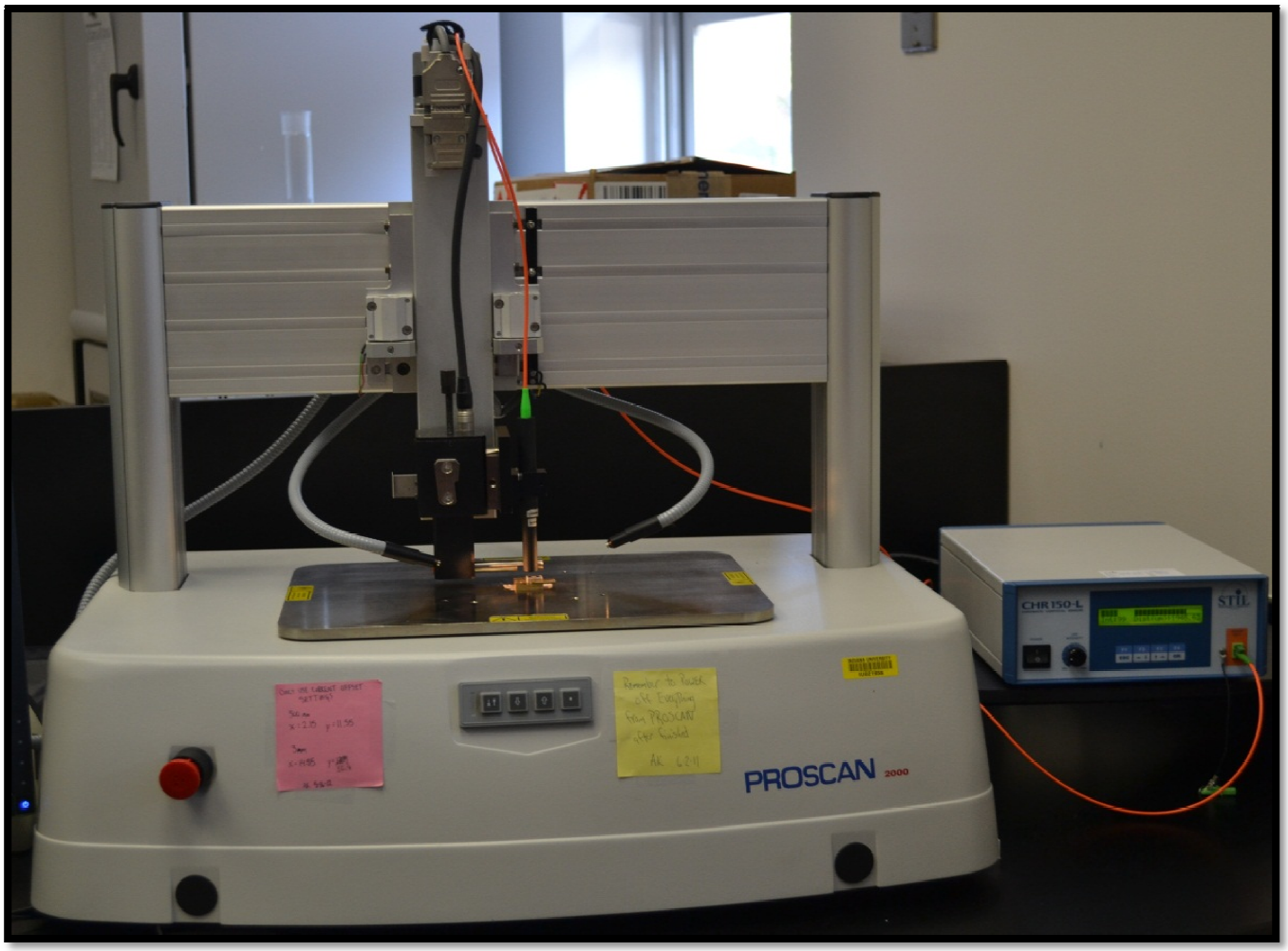
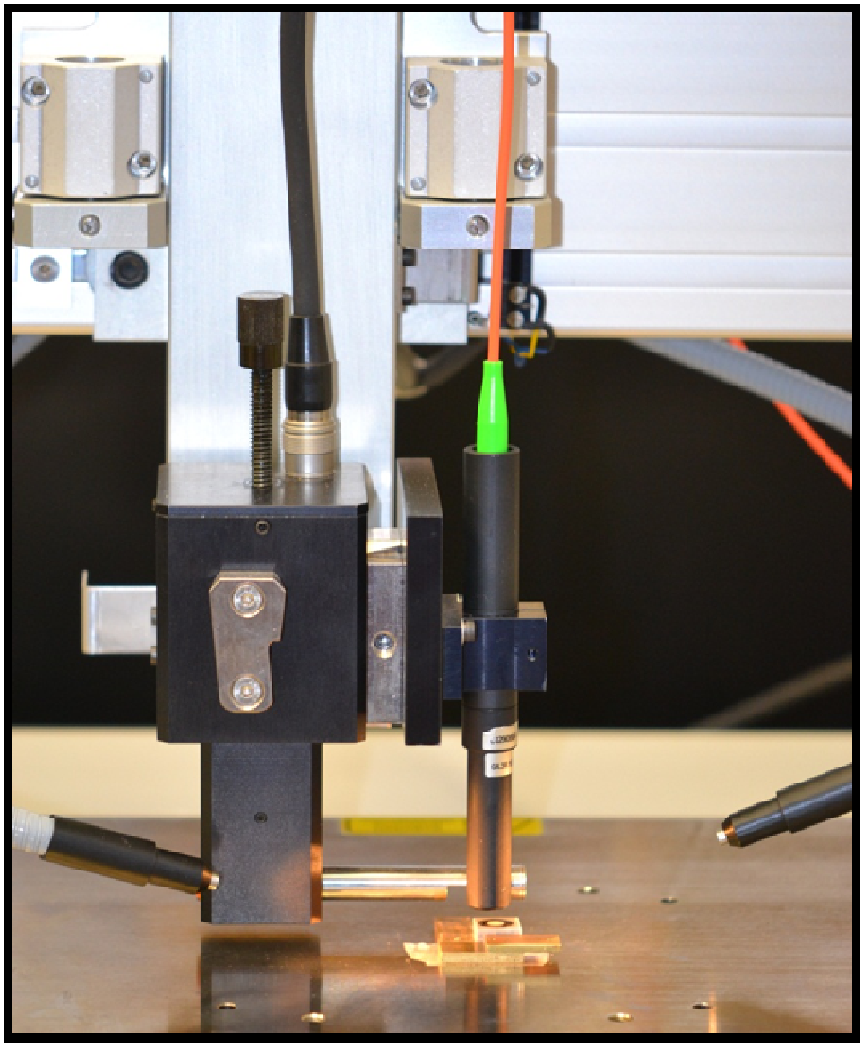


Figure 13. Specimen during the toothbrushing process.



(a)



(b)

Figure 14. a: Specimen positioned on the Proscan machine. b: Specimen during the scanning process.

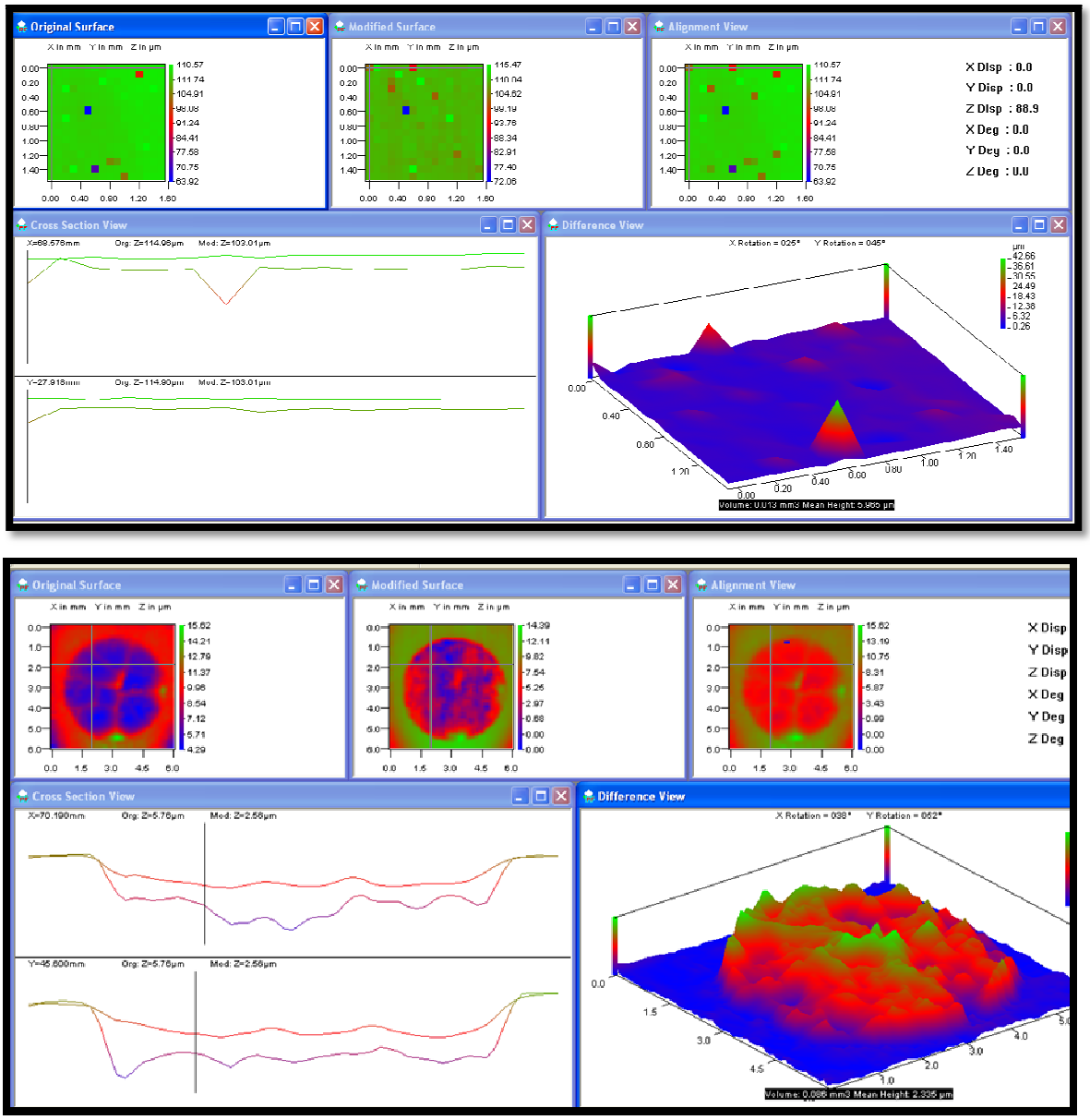
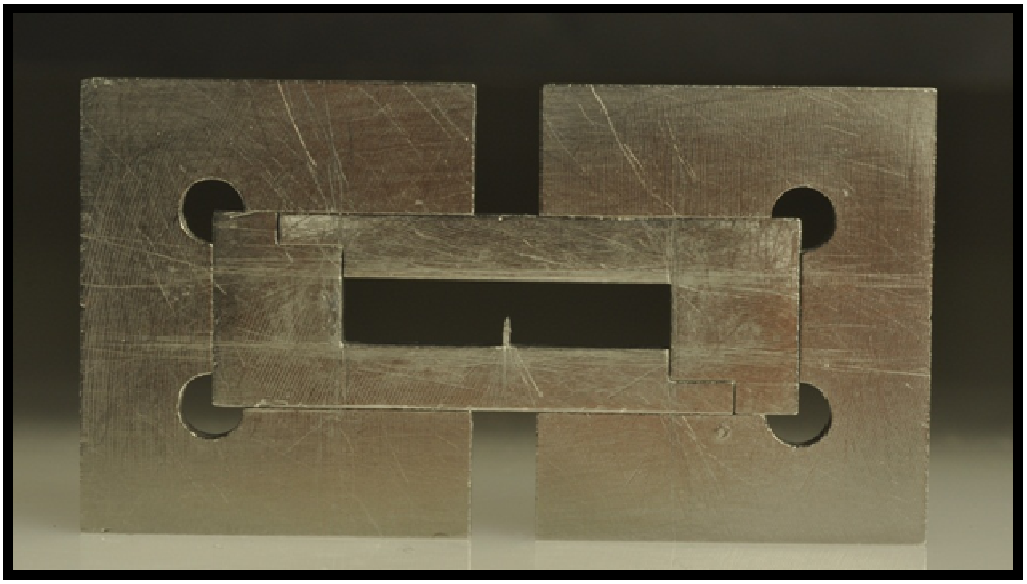


Figure 15. Material vertical loss by image subtraction (profilometer).

(a)



(b)

Figure 16. (a)(b): Mold and the mold holder for fracture toughness test.

Figure 17. MTS machine used for the fracture toughness test.

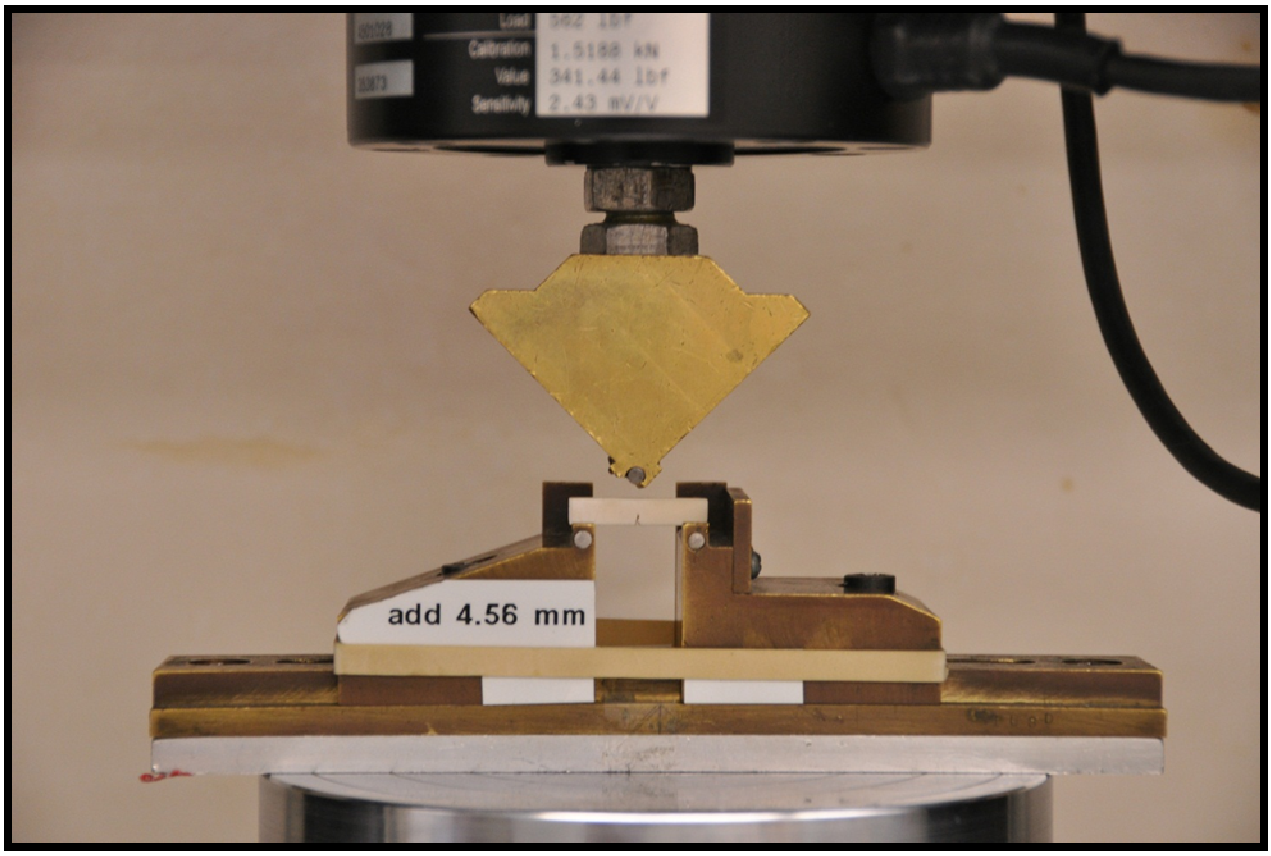
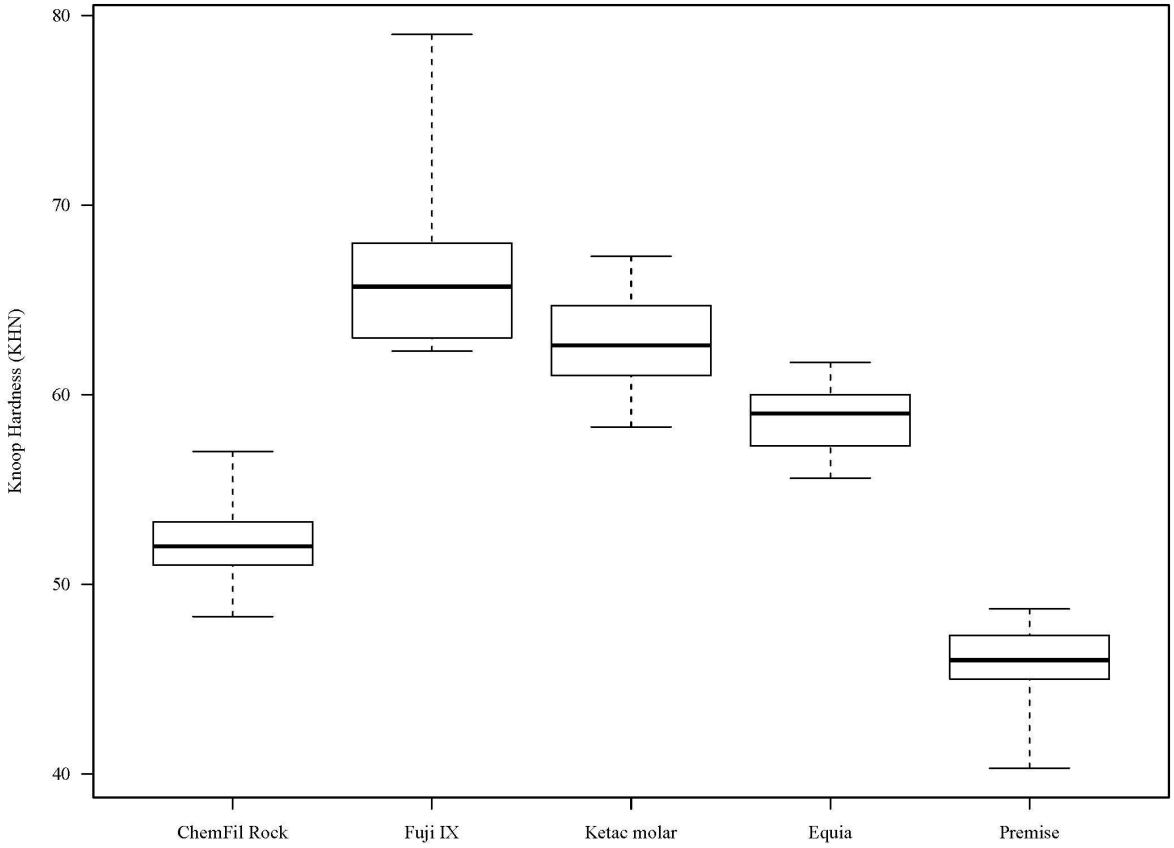


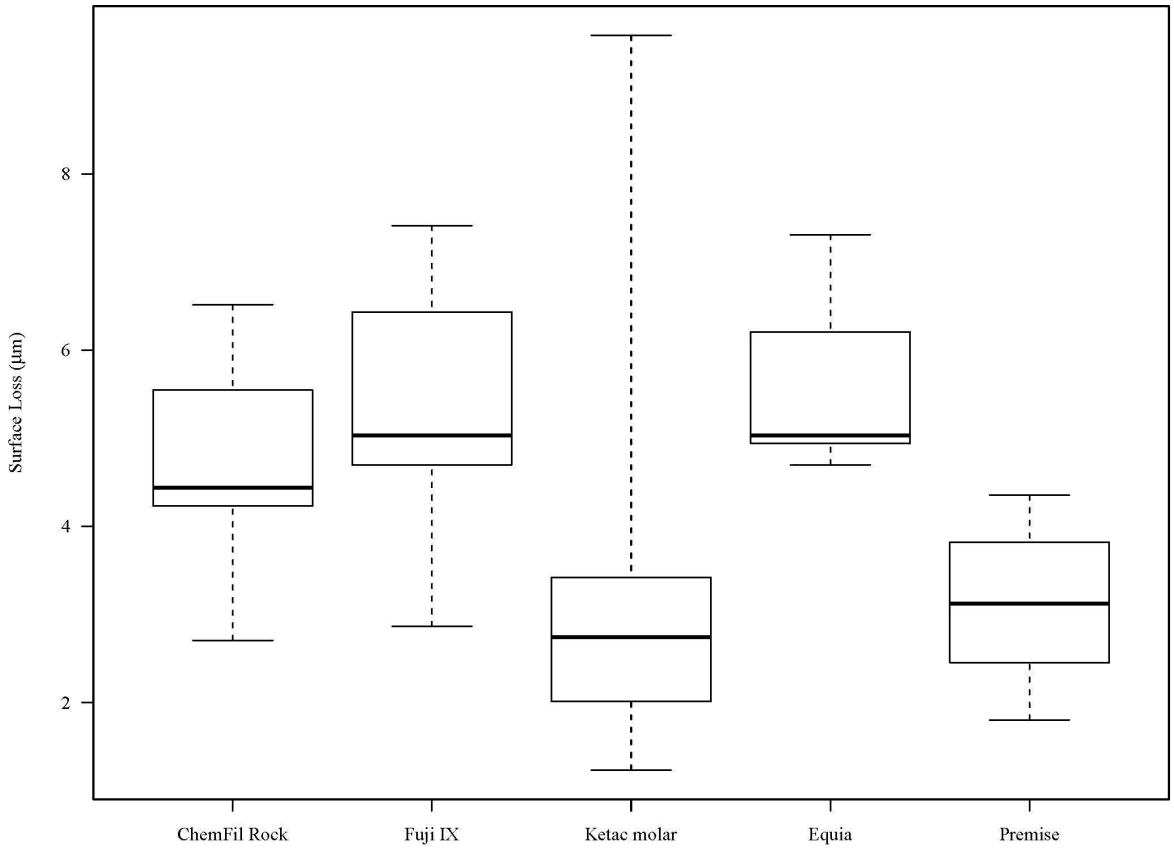
Figure 18. Specimen positioned on the MTS machine.

Figure 19. Fracture propagated at the notch after testing.



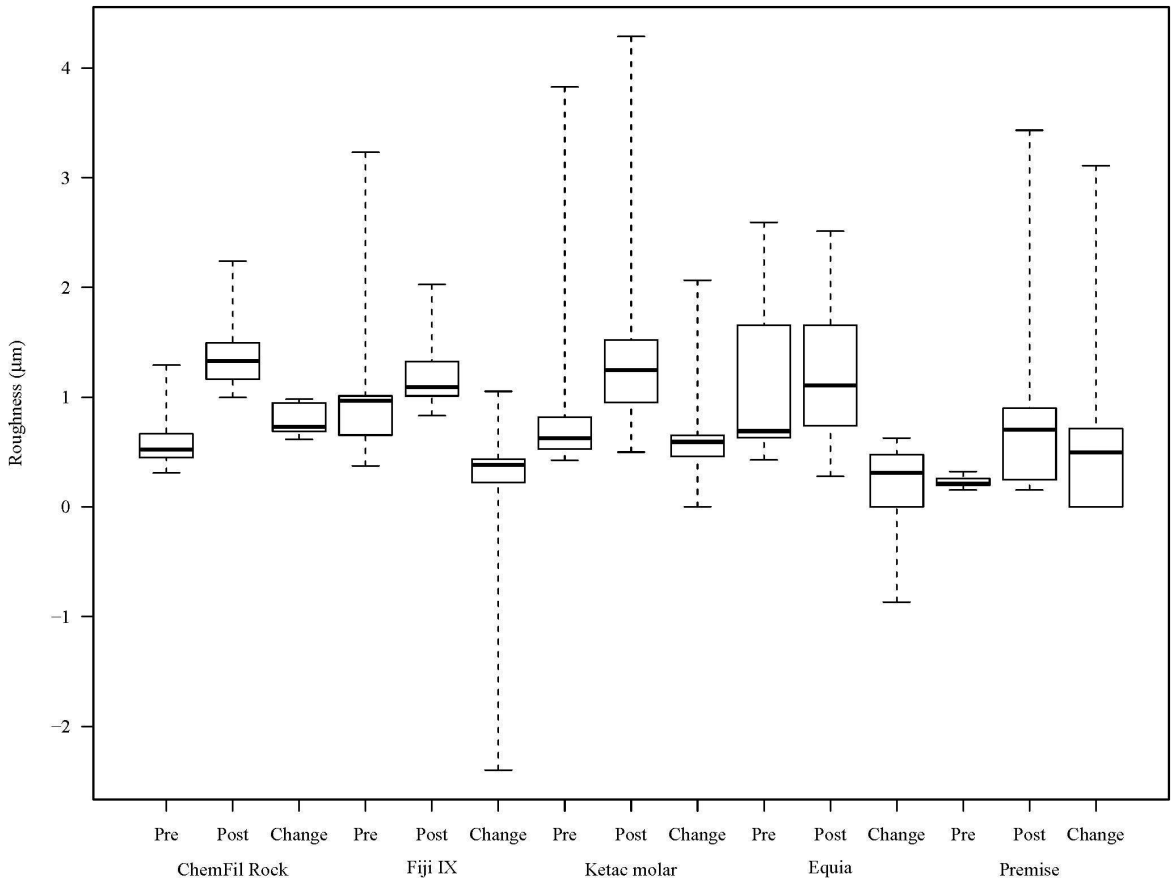
*Materials with similar letters are not statistically different.

Figure 20. Mean KHN values results and standard deviations obtained from the microhardness test.



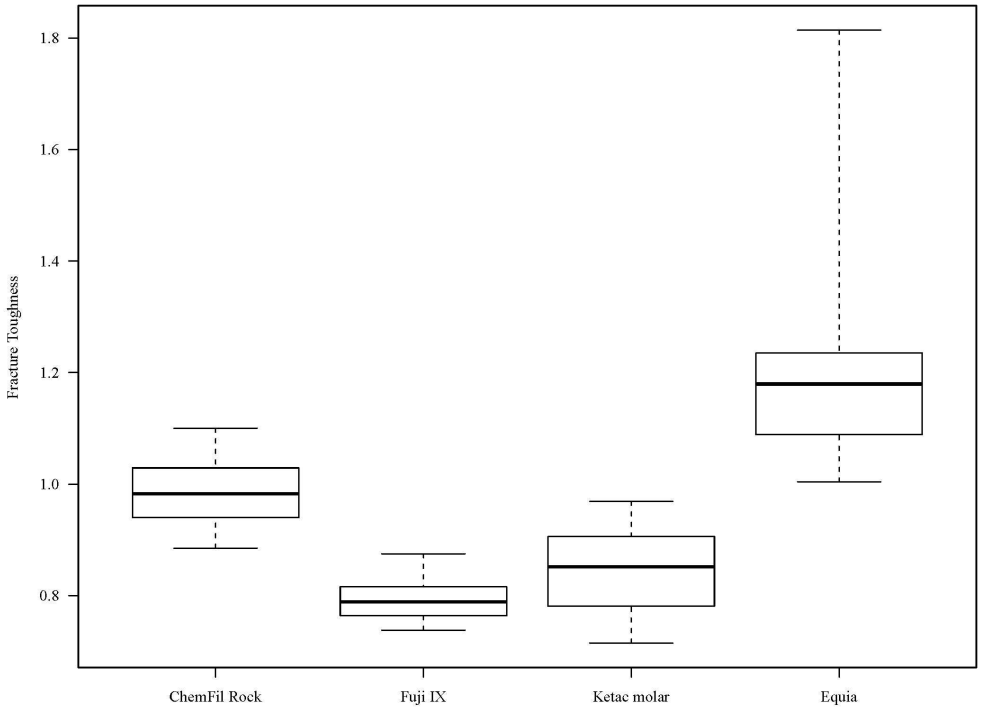
*Materials with similar letters are not statistically different.

Figure 21. Mean surface loss and standard deviation obtained from the toothbrush abrasion test.



*Materials with similar letters are not statistically different.

Figure 22. Mean roughness values results and standard deviation obtained from the profilometer machine.



*Materials with similar letters are not statistically different.

Figure 23. Mean fracture toughness values results and standard deviation obtained from MTS test machine.

DISCUSSION

GICs are often considered the restorative material of choice in many clinical situations due to their unique properties including the ability to chemically adhere to the tooth structure, fluoride release and biocompatibility.[2, 57] Although GIC has properties that meet some clinical requirements, it has some less desired physical properties which include: low wear resistance, fracture toughness and sensitivity to moisture during initial setting. [4, 5]

Manufacturers have constantly tried to improve the mechanical and physical properties of GIC to overcome its shortcoming compared to other direct restorative materials by reinforcing the material using metals such as silver amalgam alloy particles which are mixed with the glass particles or mont-morillonite clay filler, but wear-resistance was not significantly improved. [44, 45]

ChemFil Rock by Dentsply is a new glass ionomer system that has recently been introduced. The manufacturer claims that Zn incorporation in this new material has improved its mechanical properties. Including zinc as part of the glass particles enhances the setting reaction, leading to higher strength, with similar application and working times as conventional GIC.[59]

An in vitro pilot study was done before the main study for all four tests; (microhardness, abrasion, roughness and fracture toughness). The first group included three tests; (microhardness, abrasion and roughness) and there were no significant differences among the GICs, therefore, composite was added as a control group. Whereas, the second group (fracture toughness test) showed significant differences among the groups so a control group was not included in the main study.

MICROHARDNESS

Hardness is a measure of the resistance to permanent indentation on the surface. It indicates the ease of finishing of a restoration and its resistance to scratches which can compromise fatigue strength and lead to premature failure.[60] Many studies have compared the surface microhardness of different materials including GICs and composite. In this study, the microhardness load and dwell times chosen were 25 grams and 30 seconds, respectively. This was based on the literature of microhardness tests done on GIC.[2, 23, 54]

In this study the material demonstrating the highest surface microhardness was Fuji IX GP Extra with a mean value of (66.86) followed by Ketac Molar Aplicap. These results are consistent with the results from a previous study (68.7). [23] The microhardness of EQUIA Fil was significantly lower than both Fuji IX GP Extra and Ketac Molar Quick Aplicap but significantly higher than ChemFil Rock. This result is consistent with the results of a study using Vickers hardness by Zoergiebel et al.[61] where ChemFil Rock was ranked the lowest among Fuji IX GP Extra and EQUIA Fil. In the present study, the microhardness of ChemFil Rock was statistically higher than Premise (Table 2).

Composites consist mainly of two phases, i.e., filler particles surrounded by a resin matrix. The resin matrix is considered the weak phase. Filler particles vary in size and shape. Increasing the filler loading of a composite generally improves the mechanical properties of the material such as flexural strength, fracture toughness and wear resistance.[62]

The composite used in this study was Premise. It is a nanofilled composite that contains pre-polymerized filler particles.[52] Premise clearly had significantly lower microhardness values when compared to the other materials in this study. This is consistent with company claims that it has

low hardness values.[63] Also, this result is similar to other previous studies[52, 62] that show that Premise has low hardness and that pre-polymerized fillers tend to reduce microhardness values.

Based solely on the microhardness data, higher surface wear could be expected for Premise; however it showed the highest wear resistance. The microhardness results can be explained by the size of the filler particles, Premise has small filler particles (nanofilled) therefore the matrix tends to be more homogenous with the fillers, so when the Knoop hardness indenter presses against the material, it may press against the filler and the matrix resulting in a low hardness reading, whereas when the material has large size fillers (macrofilled) such as GIC, it tends to be less homogenous therefore the indenter would most probably press against the filler, resulting in a higher value. The particle size can be visualized on Figures 7, 8, 9, 10 and 11.

TOOTHBRUSH ABRASION

Toothbrush dentifrice abrasion occurs on tooth and restoration surfaces. It is more commonly seen on restorations in anterior teeth and in cervical areas.[16] Many studies have investigated toothbrush abrasion on different materials using different types of abrasives, load and number of brushing cycles.[13, 15-18] In this study we investigated the property of resistance to toothbrush abrasion for this new ZRGI material.

In general, GIC have much less wear resistance when compared with composite resins or ceramics.[64] Even though it presented the lowest surface microhardness values, the composite resin (Premise) used as a reference in this study was the most wear-resistant after toothbrushing corroborating previously published studies. [10, 12, 16, 17, 56]. This result may be related to the difference in size of the filler particles of the tested materials. Larger particles may cause higher damage to the surface as they are loosened during the wear process (shear force), leading to

three-body abrasion. While this also is expect to happen with smaller particles, the damage would be comparatively lower.

GIC display more wear compared to composites due to their different composition. Their acid-base reaction results in a matrix that consists of an ionically cross-linked polyalkenoate network which makes these cements weak when compared to the matrix of the composite which is strengthened by fillers and HEMA polymer chains.[65]

No significant differences in wear were found among the GIC tested (Table 3). This result is similar to previous studies.[46, 56]

ROUGHNESS

The surface characteristics of GICs are important since an increase in roughness might result in faster colonization and maturation of dental plaque, possibly increasing the risk of secondary caries.[13] Wear and surface roughness can also represent and predict the clinical deterioration of restorative materials.[14]

The attraction of dental plaque to roughened restorations is of serious concern. Careful polishing and finishing of dental restorations can be compromised by subsequent home care which includes toothbrushing. Most studies regarding the effect of the toothbrushing and polishing process on dental restorations have concluded that surfaces of restorations are smoother before polishing or toothbrushing and tends to increase in roughness afterwards.[24, 46, 58, 66]

In the present study, the results were based on the change in roughness values before and after the toothbrushing (average Ra) and not the actual numbers (Ra values after brushing) for

all tested materials. Composite in the study had higher mean change in surface roughness values than EQUIA Fil, Ketac Molar Quick Aplicap and Fuji IX GP Extra, yet it had no significant difference compared with the other materials.

The roughness in this study had a mean range of 0.10–0.79 μm. A comparison of the effect of toothbrushing on the GICs and the composite resin shows that Chemfil Rock had the highest significant change in the surface roughness compared to the other GIC in the study (Table 4). This result can be due to the differences in the composition of GIC. This suggests that filler particle size, shape, distribution, and number of the particles in the matrix may be responsible for these differences as previous studies have concluded.[24] The surface roughness of other materials, Ketac Molar Quick Aplicap, Fuji IX GP Extra and EQUIA Fil were not significantly different from each other.

FRACTURE TOUGHNESS

The fracture resistance of restorative materials can be characterized by the measurement of fracture toughness. Fracture toughness is the intrinsic ability of a material to resist fracture, or the amount of stress that is required to propagate a crack from a pre-existing flaw.[26] In this study we tested the materials after 24 hours since this is when the peak strength of GIC is reached.[47, 67] The results of this study showed that EQUIA Fil has the highest fracture toughness value which was statistically higher than ChemFil Rock, Ketac Molar Quick Aplicap and Fuji IX GP Extra. Both Ketac Molar Quick Aplicap and Fuji IX GP Extra were significantly lower than ChemFil Rock. The high values for EQUIA Fil may be attributed to the application of resin coating.

There were no previous studies to compare fracture toughness results for ChemFil Rock, but Ketac Molar Quick Aplicap had results similar to a previous study.[25] The other studies were done under different conditions such as: storage conditions of the tested specimens, powder liquid ratios and placing the notch in the specimens after specimen fabrication which induces stresses into the specimens[27-29, 68] resulting in fracture toughness values less than the those in this study.

Some limitations in this study need to be addressed regarding the new ZRGI. In the present study, ChemFil Rock showed good fracture toughness values relative to the other GICs. However, other mechanical properties including high surface roughness, low strength and wear resistance need to be taken into consideration. Further investigation is needed related to ChemFil Rock. Zinc has known antimicrobial activity. It would be interesting to know if the zinc incorporated into ChemFil Rock has any antibacterial effects. Also fluoride release and recharge of this new GIC need to be investigated

Within the limitations of this study, the null hypothesis was not accepted: The ZRGI did not demonstrate significantly higher fracture toughness, lower abrasive wear, lower roughness and higher surface microhardness than the three traditional GICs evaluated in this study.

SUMMARY AND CONCLUSION

The objective of this study was to compare a newly introduced Zinc- reinforced GIC to other GIC restorative materials in terms of surface microhardness, toothbrush abrasion, surface roughness and fracture toughness. For surface hardness, wear resistance and roughness tests, four GIC and one composite resin were tested. The fracture toughness test included four GIC.

Within the limitations of this study, the following conclusions can be drawn:

1. ChemFil Rock had the lowest hardness values among the GIC tested.

2. No significant difference was exhibited among GICs in toothbrush abrasion testing whereas composite was significantly more wear resistant.

3. ChemFil Rock showed higher roughness change compared to the other GIC.

4. There were significant differences in the fracture toughness test with EQUIA Fil having the highest value followed by ChemFil Rock.

Based on the results of the present study, it can be concluded that Zn incorporation in the matrix of ChemFil Rock may have increased the Fracture toughness but it didn’t play a role in improving the hardness, roughness or the wear resistance values.

Clinical significance: based on the results of this study, except for fracture toughness, ChemFil Rock does not seem to have significant advantages over other GICs.

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ABSTRACT

Mechanical properties of a new

Zinc- reinforced glass ionomer restorative material.

By

Sarah Sultan Al-Angari

Indiana University School of Dentistry

Indianapolis, Indiana

Objective: Zinc-reinforced glass ionomer restorative material (ZRGIC) has been proposed as an improved restorative material. The study compared the mechanical properties of a ZRGIC restorative material (ChemFil Rock, (Dentsply)), with three commercially available glass ionomers (GICs); Fuji IX GP Extra (GC America), Ketac Molar (3M ESPE) and EQUIA Fil (GC America). A resin composite, Premise (Kerr), was included as a control group except for fracture toughness. **Methods:** Fracture toughness (KIC) testing was done according to ISO 13586, using single edge notched-beam specimens (n=10), loaded until failure in a three-point bending test device. Specimens (n=9) for the hardness, roughness and abrasive wear testing were made by mixing and inserting the restorative materials into individual stainless steel molds followed by flattening and polishing. Knoop microhardness (KHN) was performed (25 g, 30 s),on pre-determined areas of the polished surfaces. For toothbrushing wear resistance and roughness, specimens were brushed in an automated brushing machine (200 g) with a suspension of dentifrice and water (1:1, w/v) for 20,000 strokes. Specimen surfaces were scanned in an optical profilometer before and after brushing to obtain surface roughness (Ra) and mean height (surface) loss using image subtraction and dedicated software. Data were analyzed using Wilcoxon Rank Sum tests (α=0.05). **Results:** ChemFil Rock had the highest change in surface roughness (Ra)(0.79±0.14; $p<0.001$) and the lowest microhardness (KHN) values (52.39±2.67; $p<0.05$) among all GICs. Its wear resistance was comparable to other GICs ($p>0.05$). ChemFil Rock had lower fracture toughness (0.99±0.07, KIC) compared to Equia Fil ($p<0.01$) and higher compared to the other GICs ($p<0.01$). **Conclusion:** The new ZRGIC restorative

material showed intermediate fracture toughness, high change in surface roughness, and low microhardness compared to three other commercial GICs. All materials were supplied by respective manufacturers.

CURRICULUM VITAE

Sarah Sultan Al-Angari

September 1983	Born in Jeddah, Saudi Arabia
June 2001	High School Diploma Dar El-Eloum High School Riyadh, Saudi Arabia
July 2001 to July 2007	Bachelor of Dental Surgery (BDS) College of Dentistry, King Saud University, Riyadh, Saudi Arabia.
July 2007 to July 2008	Dental Internship College of Dentistry, King Saud University, Riyadh, Saudi Arabia.
July 2008 to present	Demonstrator (Teaching Assistant) Operative Dentistry Division, Department of Restorative Dentistry, College of Dentistry, King Saud University Riyadh, Saudi Arabia.
August 2010 to July 2012	Operative Dentistry Graduate Program, Indiana University School of Dentistry, Indianapolis, Indiana.
August 2012	Accepted by doctoral program in Dental Science Indiana University School of Dentistry Indianapolis, IN

Professional Organizations

The Academy of Operative Dentistry
Saudi Dental Society
The Saudi Commission for Health Specialties